



Calhoun: The NPS Institutional Archive
DSpace Repository

Theses and Dissertations

1. Thesis and Dissertation Collection, all items

1960

Investigation of the effect of simultaneous
plastic strain on the recovery and
recrystallization behavior of a cold-worked
vacuum-melted high purity iron

Gurney, Sumner

<http://hdl.handle.net/10945/12422>

This publication is a work of the U.S. Government as defined in Title 17, United States Code, Section 101. Copyright protection is not available for this work in the United States.

Downloaded from NPS Archive: Calhoun



Calhoun is the Naval Postgraduate School's public access digital repository for research materials and institutional publications created by the NPS community. Calhoun is named for Professor of Mathematics Guy K. Calhoun, NPS's first appointed -- and published -- scholarly author.

Dudley Knox Library / Naval Postgraduate School
411 Dyer Road / 1 University Circle
Monterey, California USA 93943

<http://www.nps.edu/library>

NPS ARCHIVE
1960
GURNEY, S.

INVESTIGATION OF THE EFFECT OF SIMULTANEOUS
PLASTIC STRAIN ON THE RECOVERY AND
RECRYSTALLIZATION BEHAVIOR OF A
COLD-WORKED VACUUM-MELTED
HIGH PURITY IRON
SUMNER GURNEY

LIBRARY
U.S. NAVAL POSTGRADUATE SCHOOL
MONTEREY, CALIFORNIA

UNITED STATES NAVAL POSTGRADUATE SCHOOL



THESIS

INVESTIGATION OF THE EFFECT
OF
SIMULTANEOUS PLASTIC STRAIN
ON THE
RECOVERY AND RECRYSTALLIZATION BEHAVIOR
OF A
COLD-WORKED VACUUM-MELTED HIGH PURITY IRON
by
Sumner Gurney
Lieutenant Commander, United States Navy

INVESTIGATION OF THE EFFECT
OF
SIMULTANEOUS PLASTIC STRAIN
ON THE
RECOVERY AND RECRYSTALLIZATION BEHAVIOR
OF A
COLD-WORKED VACUUM-MELTED HIGH PURITY IRON

* * * * *

Sumner Gurney

INVESTIGATION OF THE EFFECT
OF
SIMULTANEOUS PLASTIC STRAIN
ON THE
RECOVERY AND RECRYSTALLIZATION BEHAVIOR
OF A
COLD-WORKED VACUUM-MELTED HIGH PURITY IRON

by

Sumner Gurney

//

Lieutenant Commander , United States Navy

Submitted in partial fulfillment of
the requirements for the degree of

MASTER OF SCIENCE
IN
CHEMISTRY

United States Naval Postgraduate School
Monterey , California

1960

TYPE ARCHIVE
1960
GURNEY, S.

~~Thesis~~
~~6-867~~

DUDLEY KNOX LIBRARY
NAVAL POSTGRADUATE SCHOOL
MONTEREY, CA 93943-5101

INVESTIGATION OF THE EFFECT

OF

SIMULTANEOUS PLASTIC STRAIN

ON THE

RECOVERY AND RECRYSTALLIZATION BEHAVIOR

OF A

COLD-WORKED VACUUM-MELTED HIGH PURITY IRON

by

Sumner Gurney

This work is accepted as fulfilling
the thesis requirements for the degree of

MASTER OF SCIENCE

IN

CHEMISTRY

from the

United States Naval Postgraduate School

ABSTRACT

This thesis reports an investigation of the effect of simultaneous plastic strain on the recovery and recrystallization behavior of a vacuum-melted high purity iron cold-rolled 60% from the as received hot-rolled condition. Thyatron-controlled constant-strain-rate motor units were utilized for application of a tensile load to conventional tensile sheet specimens which were heated in constant temperature salt baths.

Rockwell-30T hardness measurements and microstructural studies were used as the means of following the progress of recovery and recrystallization of static (conventional) and dynamic (superimposed strain) specimens during annealing.

The softening behavior of both static and dynamic anneals were modified by the presence of polygonization which competed with the normally expected recrystallization process. This behavior becomes more pronounced with decrease in temperature and/or increase of simultaneous superimposed strain rate.

TABLE OF CONTENTS

Section	Title	Page
1.	Introduction	1
2.	Test Material	4
3.	Experimental Procedure	7
	A. Static Study Procedure	7
	B. Dynamic Study Procedure	8
4.	Experimental Results	13
	A. Static Study Results	13
	B. Dynamic Study Results	23
5.	Summary of Results and Conclusions	36
6.	Acknowledgements	37
7.	Bibliography	38

LIST OF ILLUSTRATIONS

Figure		Page
1.	Experimental Apparatus	10
2.	Constant Strain Rate Unit	11
3.	Tensile Sheet Specimen Used for Dynamic Strain	12
4.	The Effect of Annealing Time at Various Temperatures on the Room Temperature Hardness for a 60% Cold-Rolled Vacuum-Melted High Purity Iron	18
5.	As Rolled Condition (Photomicrograph)	19
6.	At 540°C for One Minute (Photomicrograph)	19
7.	At 540°C for Six Minutes (Photomicrograph)	19
8.	At 540°C for 18 Minutes (Photomicrograph)	19
9.	At 540°C for 42 Minutes (Photomicrograph)	20
10.	At 540°C for Three Hours (Photomicrograph)	20
11.	At 540°C for 30 Hours (Photomicrograph)	20
12.	At 540°C for 329.6 Hours (Photomicrograph)	20
13.	At 597°C for Two Minutes (Photomicrograph)	21
14.	At 540°C for 42 Minutes (Photomicrograph)	21
15.	At 482°C for 96 Hours (Photomicrograph)	21
16.	At 448.5°C for 1242 Hours (Photomicrograph)	21
17.	At 597°C for Two Minutes (Photomicrograph)	22
18.	At 448.5°C for 1242 Hours (Photomicrograph)	22
19.	The Effect of Simultaneous Strain on the Softening Behavior of a 60% Cold-Rolled Vacuum-Melted High Purity Iron	29
20.	At 540°C for 18 Minutes (Photomicrograph)	30

LIST OF ILLUSTRATIONS

Figure		Page
21.	At 540°C for Three Hours (Photomicrograph)	30
22.	At 540°C for 30 Hours (Photomicrograph)	30
23.	At 540°C, Under .6%/HR. Strain Rate, for 18 Minutes (Photomicrograph)	31
24.	At 540°C, Under .6%/HR. Strain Rate, for 3 Hours (Photomicrograph)	31
25.	At 540°C, Under .6%/HR. Strain Rate, for 30 Hours (Photomicrograph)	31
26.	At 540°C for 30 Hours (Photomicrograph)	32
27.	At 540°C, Under .0005%/HR. Strain Rate, for 30 Hours (Photomicrograph)	32
28.	At 540°C, Under .6%/HR. Strain Rate, for 30 Hours (Photomicrograph)	32
29.	At 482°C for 1.5 Hours (Photomicrograph)	33
30.	At 482°C for 11.7 Hours (Photomicrograph)	33
31.	At 482°C for 49 Hours (Photomicrograph)	33
32.	At 482°C, Under .6%/HR. Strain Rate, for 1.5 Hours (Photomicrograph)	34
33.	At 482°C, Under .6%/HR. Strain Rate, for 11.7 Hours (Photomicrograph)	34
34.	At 482°C, Under .6%/HR. Strain Rate, for 49 Hours (Photomicrograph)	34
35.	At 482°C for 49 Hours (Photomicrograph)	35
36.	At 482°C, Under .005%/HR. Strain Rate, for 49 Hours (Photomicrograph)	35
37.	At 482°C, Under .6%/HR. Strain Rate, for 49 Hours (Photomicrograph)	35

1. Introduction

There are many ways that metals and alloys can be strengthened, one of which is cold working; but, a cold-worked metal is subject to the softening of recovery and recrystallization at elevated temperatures. The upper temperature limit to which a metal may be heated and still retain its cold-worked properties is generally accepted as the minimum temperature at which softening takes place as determined by the static annealing of the cold-worked state. However, in practice one must consider the possible effects of simultaneous plastic strain and stress, imposed by the existing load, on modifying the softening behavior as obtained by static tests. Thus, if the softening is accelerated, a metal which would be expected to exhibit stability under static conditions (no load) may be unstable when subjected to dynamic conditions (under load) at the same temperatures.

Cold working a metal alters the distribution of line and point imperfections already existing in the unworked metal and generates additional imperfections. The effects of cold work are not uniformly distributed throughout the metal and it is this resultant inhomogeneity that affects the mechanisms of the various restoration processes.

Fundamentally, the restoration of the metal's original properties may be brought about by the mechanisms of recovery, polygonization, and/or recrystallization. The driving force for all three processes basically results from the stored energy of cold work. Each process is time and temperature dependent with the stored energy of cold work

serving to reduce the thermal activation energy required for the process .

Recovery is generally defined as the change in cold-worked properties without noticeable microstructural changes . It takes place through changes in the arrangement and number of point defects and dislocations .

Polygonization is a restoration mechanism whereby edge dislocations , prompted by thermal activation , glide along their respective slip planes and climb in a direction ninety degrees from the original slip plane as a result of the dislocation and vacancy interaction . This glide and climb mechanism results in the dislocation grouping being changed from horizontal to vertical . The newly formed vertical subgrain boundaries join others from other slip planes by Y-junction formation and subsequently coalesce into subgrained polygon structures .

Recrystallization is a process of nucleation and growth . In this process strain-free nuclei are formed from the cold-worked matrix and subsequently grow by consuming the original strained grains . Recrystallization generally removes the property changes of cold work that the prior recovery and polygonization have not eliminated .

The technical literature evidences considerable research and experimental effort toward a better understanding of the recovery and recrystallization kinetics of cold-worked metals and alloys subjected to various annealing temperatures . (1-2) However , little effort has been expended in the investigation of the effect of simultaneous strain on the recovery and recrystallization behavior . Reference cited by the literature

in this regard has been generally incidental to studies on creep. (3) Nothing has been uncovered by this author's literature research in regard to studies primarily directed toward a systematic investigation on the interrelation between dynamic and static softening behavior of a cold-worked metal.

Sherby, Goldberg and Dorn, in studying the recovery behavior of a high purity aluminum prestrained 15% at 78°K, found that the application of a creep load at the annealing temperature accelerated the recovery rate. (4) Grant and Bucklin investigating creep behavior of a cold worked monel noted that "new grains" appeared during creep at temperatures considerably below the static recrystallization temperature for the same time interval. (5) This information, coupled with the considerable amount in regard to static recovery and recrystallization, intuitively suggests the need for further investigation of the effect of dynamic loading on cold-worked metals undergoing softening at elevated temperatures.

Thus, it was decided to study the comparison of the softening behavior of a cold-worked high purity iron under static and dynamic annealing conditions. The term "dynamic" used throughout this report implies that the cold-worked metal was subjected to a constant strain rate during the anneal. The term "static" implies softening under no-load conditions. Though polygonization is generally considered as a type of "recovery" process, due to its formation of subgrains within the original deformed grains, it will be noted here as a distinct process and discussed accordingly in this report.

2. Test Material

A vacuum-melted "super purity" electrolytic iron provided by the Ford Motor Company was used as the test material. Two strips of iron, each approximately 1/4 inch by 3 inch by 6 feet in length were received from the supplier. One piece was labelled C65A and its reported analysis was .003% carbon and .003% residual oxygen. Ford Motor Company stated that this piece exhibited discontinuous grain growth with attendant irregular grain structure, probably due to excess oxygen. The other piece supplied, C66A, was reported to have .02% carbon with no attendant anomalies. Each piece of iron was received in the hot-rolled and pickled condition.

A five-inch piece was cut from the end of each bar for subsequent rolling to 30% reduction in thickness. The 30%-reduced strips ($.170 \pm .001$ inch thickness) were then cut into 3/8-inch by 3/4-inch specimens. Specimens from each bar were then subjected to 597°C salt bath temperatures for periods of one to 94 hours and subsequent hardness and microstructure comparisons made. The molten salt bath used consisted of a ternary carbonate eutectic with a melting temperature of 440°C. It was found that both types recrystallized prior to 48 hours. The C66A iron was softened from a Superficial Rockwell 30T hardness of 69 (30% cold rolled) to a value of 39 after 94 hours, at which point it was completely recrystallized and grain growth was in evidence. The C65A iron suffered a similar reduction in hardness from 66 to 30 over the same time period. Microstructural studies of the two different iron specimens were

made after 597°C salt bath subjection times of one, four, 20, 48, 73, and 94 hours. It was decided on the basis of these microstructural studies that the purer iron (C65A), which was slightly softer than the C66A, did not exhibit any more discontinuous grain growth and irregular structure than the C66A iron and, thus, was chosen as the bar for further dynamic and static comparison studies. In fact, both bars showed normal recrystallized structures. A recrystallized grain size of 16 grains per mm. length was obtained for the C65A after the 48 hour anneal.

The original C65A iron bar (C=.003, O=.003) was recut and reduced in thickness from $.249 \pm .003$ to $.1706 \pm .0004$ inches (30% reduction) by repeated rolling. The effect of the number of passes through the rolling mill to arrive at a certain per cent reduction was investigated and found to be immaterial as the hardness was the same whether many or few passes were made. The 30%-reduced bar was then sheared into nine by one-inch strips and recrystallized by subjection to a 597°C salt bath temperature for a period of 48 hours. These strips were subsequently reduced from $.1706 \pm .0004$ to $.0680 \pm .0004$ inch thickness, resulting in the iron having a 60% cold-work reduction in thickness by rolling. After this final rolling, the 60% cold-worked strips were sheared into 3/4-inch by 3/8-inch specimens for the static recrystallization study. Other strips were fabricated into specimens for the dynamic study and are described later.

Due to the limited thickness of the static and dynamic specimens the Superficial Rockwell Hardness Scale was used in all cases and will

be referred to as Rockwell 30T in this report. The average Rockwell 30T hardness of the 60% cold-worked dynamic and static specimens was 70 ± 1 .

3. Experimental Procedure

A. Static Study Procedure

The static recrystallization study was made using molten salt baths for the isothermal annealing medium. The salt used was Houghton Liquid Heat No. 235, a ternary eutectic composition of lithium, potassium, and sodium carbonates with a melting temperature of 440°C and an effective stability range of 445°C to 920°C . Molten salt baths were used instead of air furnaces due to the high temperature range selected for the study, 448.5°C to 597°C , in order to minimize and control the time required to reach test temperatures. Use of salt baths, regenerated and cleaned of oxygen periodically by CO_2 addition, also precludes undue oxidation of specimens. Motor-driven stirrers were used in the baths, resulting in a temperature variation of less than 1°C within a working volume of seven inches depth by seven inches diameter. Temperature control was maintained within $\pm 1^{\circ}\text{C}$ of the reported temperature for the test times.

It was found that the salt had no deleterious effect on the hardness of specimen surfaces. The solidified salt was removed from the cooled specimens by immersion in a 5% acetic acid solution over a period of two hours. A minimum of four hardness readings was taken per specimen and the values averaged.

In this static study the progress of recovery and recrystallization of the 60% cold-worked material was followed by changes in microstructure and Rockwell 30T hardness measurements. The salt bath

temperatures of the static study were 448.5°C , 482°C , 510°C , 540°C , 579°C , and 597°C . The lower temperature was dictated by the freezing point of the salt used. The upper temperature was limited by the rapidity of recrystallization. The time for complete recrystallization at the upper temperature limit was found to be 15 minutes.

B. Dynamic Study Procedure

Similar salt baths and temperature control equipment were used for both dynamic and static studies. An overall view of the dynamic experimental apparatus is shown in Fig. 1. The tensile load is transmitted to the specimen (see Fig. 3) by a stainless steel cage contained in the salt bath. The bottom of the specimen, which is mounted on the extensometer, on being immersed into the bath is hooked to the bottom of the cage; the specimen is then contained within the center of the seven-inch uniform temperature depth.

The load-bearing constant-strain-rate components were installed on existing creep units. The creep lever arm was replaced by a cam and yoke arrangement in each of the units as shown in Fig. 2. The extensometer, containing the dynamic specimen, was attached by a unidirectional mechanical linkage to the yoke whose solely vertical movement was a result of its traverse of the cam perimeter. The cam was rotated by a $1/8\text{-H.P.}$, 115-volt DC shunt-wound motor through two reducing-gear stages, each of 1700:1; the motor speed at the cam being reduced by 2.89×10^6 of its value. The output speed of the motor was maintained constant by a RLC thyatron control circuit whose use was to

control the motor armature current for constant speed operation during motor load fluctuations imposed by the strained specimens. The motor field current was maintained constant by supply voltage regulation. By adjusting one motor speed to a minimum and the other to a maximum value, constant strain rates of .005% and .6% per hour, respectively, were obtained. The strain in the specimen was checked by a dial guage having .0001-inch divisions.

The progress of the dynamic study runs was followed by Rockwell 30T hardness measurements and microstructural studies.

Many types and combinations of etchants were experimented with to determine one suitable for microscopic examination of the various test specimens. As a result of this experimentation, it was decided to immerse the specimens in a solution containing two parts by volume of saturated picric acid in ethyl alcohol for 20 seconds followed by water and alcohol rinses and then a ten-second immersion in a solution of one part concentrated HNO_3 to 99 parts ethyl alcohol.

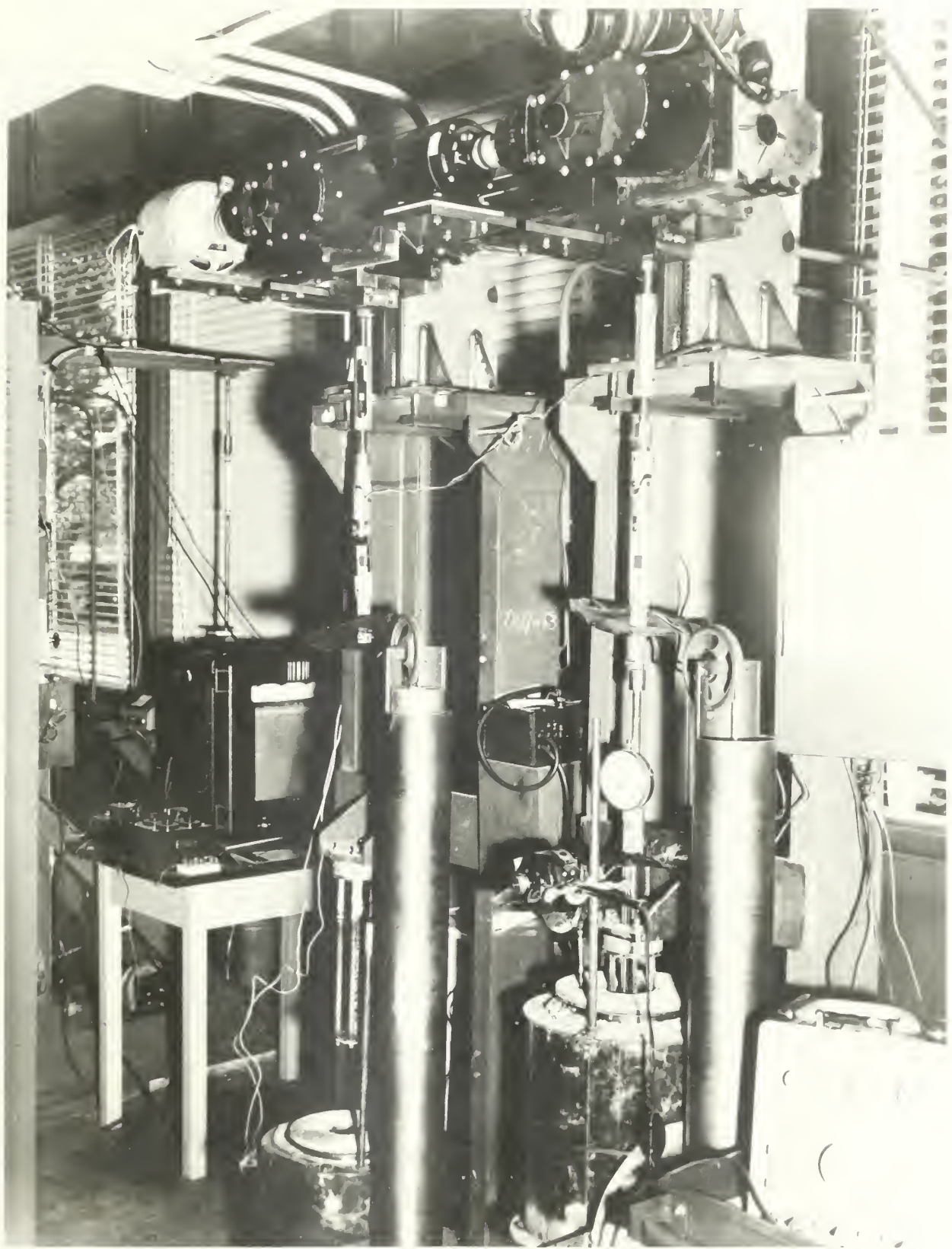


FIG.1 EXPERIMENTAL APPARATUS

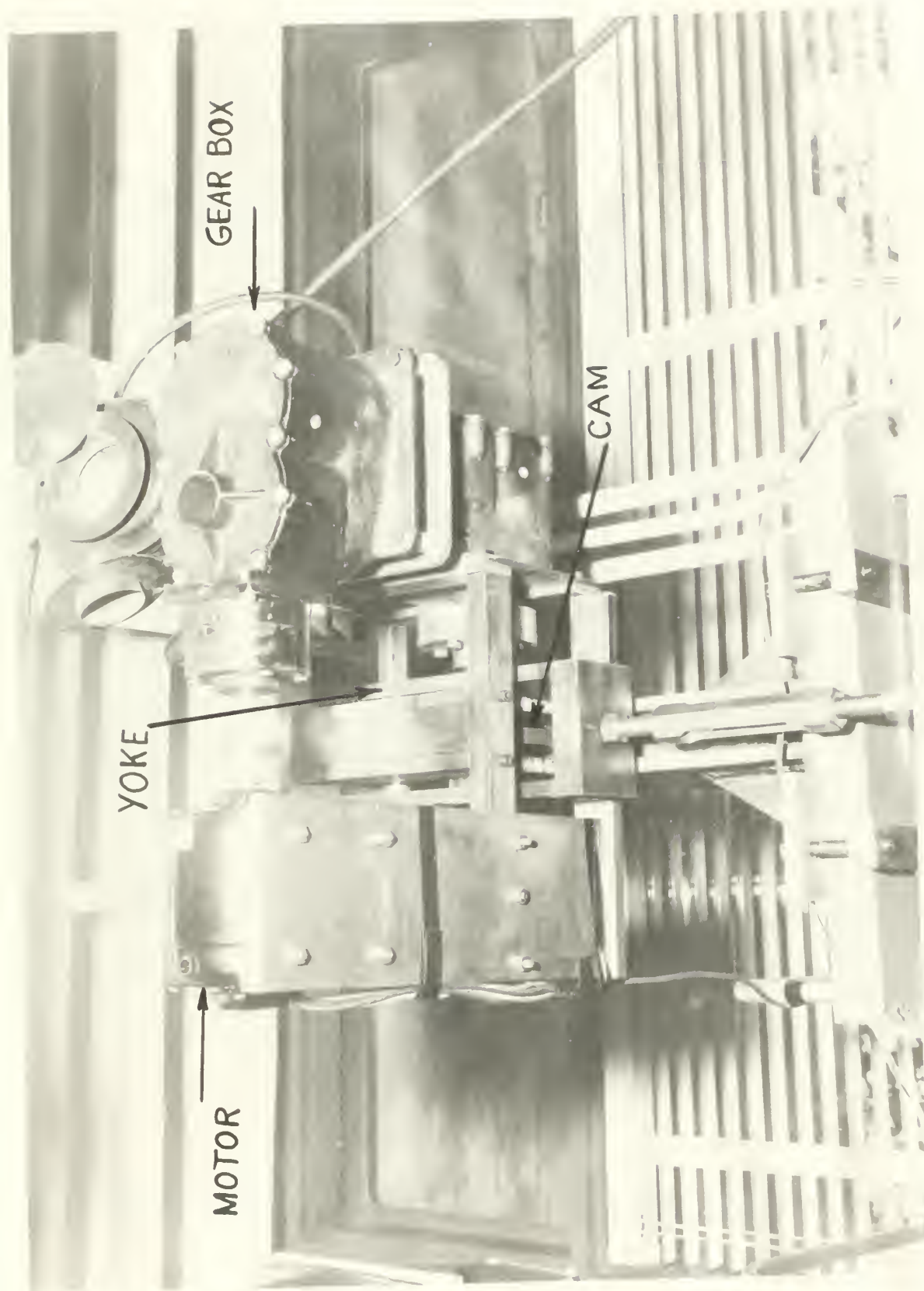


FIG. 2 CONSTANT STRAIN RATE UNIT

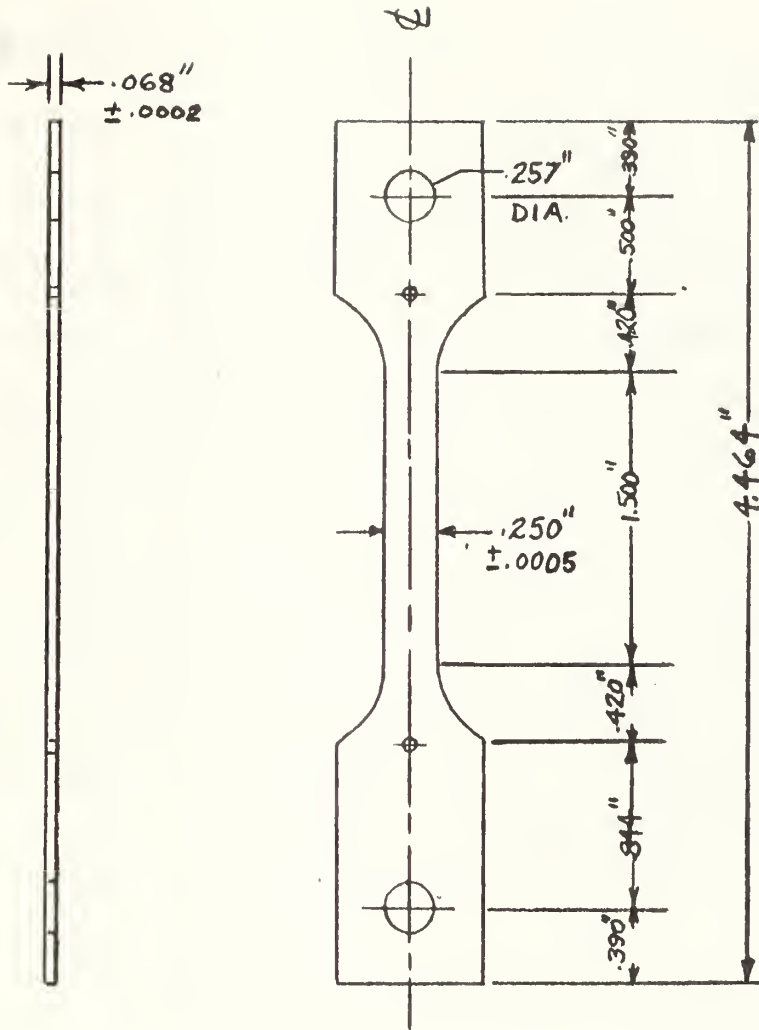


FIG. 3
TENSILE SHEET SPECIMEN USED FOR DYNAMIC STRAIN

4. Experimental Results

A. Static Study Results

Fig. 4 shows the annealing curves obtained at temperatures of 448.5°C , 482°C , 510°C , 540°C , 579°C , and 597°C for the 60% cold-rolled vacuum-melted high purity iron whose room temperature hardness is plotted as a function of logarithmic time. Such curves generally depict three distinct regions; namely an initial slow drop in hardness followed by a sudden rapid drop which terminates sharply into a final continual slow decrease in hardness. The three regions represent recovery, recrystallization and grain growth, respectively. The same characteristics are obtained when hardness is plotted as a function of temperature with time constant. With temperature as a variable, it has been reported for iron that the intermediate region corresponded completely to microstructural recrystallization. (6) An investigation now being undertaken on a low purity iron (7) again shows this typical behavior when the hardness is plotted as a function of logarithmic time at a series of temperatures.

With reference to Fig. 4 the three regions, somewhat typical of recovery, recrystallization, and grain growth, are apparent; however, in contrast to the above reported literature results, the change from one region to another is gradual. Furthermore, both the relative drop in hardness in the apparent recovery region is considerably greater and the time period during which the rapid drop in hardness takes place is about ten times longer than obtained for the low purity iron. (7) The

low purity iron was examined for 50% and 75% cold-rolled states.

In an attempt to understand these differences and to determine the degree of parallelism between microstructural and hardness changes, microstructural observations were made on a series of specimens annealed at 540°C . This temperature corresponds to the curve having all three regions. Figs. 5 through 12 show photomicrographs of these microstructures taken at a magnification of 150.

The photomicrographs clearly show that recrystallization was the predominant microstructural change in progress as the hardness decreased with time-at-temperature. It is interesting to note, however, that the deformation bands clearly evident in the as-cold-rolled specimen become somewhat diffuse after 1 minute at 540°C . Additional time at temperature does not bring about any further visual change in the unrecrystallized deformed grain. The deformation bands result from the accumulation of dislocations and represent zones of high distortion and plastic curvature separating less severely strained disoriented regions. The thermal energy superimposed on the high residual back stresses of the piled-up dislocations presumably bring about an almost instantaneous glide of these dislocations back toward the less severely strained regions, causing a widening or decrease in sharpness of the deformation bands. Subsequent elimination (or decrease) in the residual strain energy (as suggested by the changing microstructures) takes place by recrystallization which is accompanied and followed by some grain growth.

It is quite apparent from the microstructures (Figs. 6 and 7)

that recrystallization occurs in the so-called recovery region. In fact, as may be seen from Table 1, the relative drop in hardness may be attributed almost entirely to recrystallization.

Table 1

Data Showing Correlation of Hardness Change and Recrystallized Area

Time at 540 ^o C	Estimated % of Area Recrystallized	% Rockwell 30T Hardness Decrease *
1 minute	2	2.2
6 minutes	11	11.1
18 minutes	30	28.8
42 minutes	50	48.9
3 hours	81	78.4
30 hours	99	98.2
100 hours	100	100.0

* $70-H_t/70-33 \times 100$ Where H_t is the hardness at time t and 33 corresponds to the hardness when the material is completely recrystallized.

A parallelism between hardness drop and the degree of recrystallization occurs only if one considers that the apparent recovery region is, in fact, part of the recrystallization region.

The shape of this entire recrystallization curve at 540^oC (prior to grain growth) could well be the result of the elimination of high-strain-energy nucleus regions by some competing process, the effect of which lessens with increasing recrystallization. If this occurs, due to the exponential temperature dependence on nucleation (2) it would be expected that the recrystallization would be more severely hampered at the

lower temperatures. To ascertain this, microstructures of a partially recrystallized state were examined for the various temperatures.

Figs. 13 through 16 are photomicrographs at 150 magnification of static specimens softened approximately 50% at the different annealing temperatures of 597°C , 540°C , 482°C , and 448.5°C . The photomicrographs clearly evidence that, even for the same hardness change, recrystallization was much more predominant in the 597°C specimen microstructure than in that annealed at 448.5°C . The degree of recrystallization, thus, was shown to decrease with decreasing temperature, as indicated by the amount of new strain-free grain formation, for the same corresponding hardness values. As it is generally accepted that recovery alone is responsible for only slight decreases in hardness, it is evident that some other restorative phenomena such as polygonization must account for the observation that, though at the same reduction in hardness, the 540°C specimen exhibited almost 50% less recrystallization than the specimen at 597°C . Figs. 17 and 18 are photomicrographs of the same characteristic microstructures of Figs. 13 and 16, but at 2000 magnification. The clear recrystallized grains of Fig. 17 stand out nicely between the two original strained grains. There is no polygonization evident in the strained grains, just the typical deformation bands of cold work. Fig. 18, however, clearly shows a polygonized substructure in the deformed grains surrounding a recrystallized grain. It is also noted that the line A-A along the boundary between two strained grains in Fig. 17 is regular and almost uniform, typical of nonpolygonized adjacent

strained-grain boundaries; whereas in Fig. 18 the line B-B denoting the same type boundary is irregular due to the polygonized substructures. This irregularity is typical of adjacent polygonized grains and substantiated in the literature. (8) Despite the polygonization in evidence in Fig. 18, Figs. 13 through 16 still show that recrystallization was the predominant factor in the softening of the test material, especially at the higher temperature static anneals.

The competition between polygonization and recrystallization evidenced in the aforesaid discussion is substantiated by Talbot's work in France on the polygonization of zone-refined iron (9) during which he found that the purer the iron, the greater the ease with which polygonized substructures can form and the lower the polygonization temperature. Though it is recognized, from Burke and Turnbull's summary of the qualitative laws of recrystallization (2), that increase of anneal time decreases the temperature necessary for recrystallization, it is evident that Figs. 13 through 18 show some grains have recovered by polygonization, leaving no driving force to promote recrystallization of those grains. The decrease in anneal temperature has favored polygonization in these grains in preference to recrystallization and thereby modifying the softening curves. At higher temperatures, presumably, an attempt at polygonization, which was evidently effectively interrupted by recrystallization, again was responsible for modification of these curves. The differences between these curves and those obtained for the impure iron, thus, are consistent with this interpretation and the work by Talbot (9).

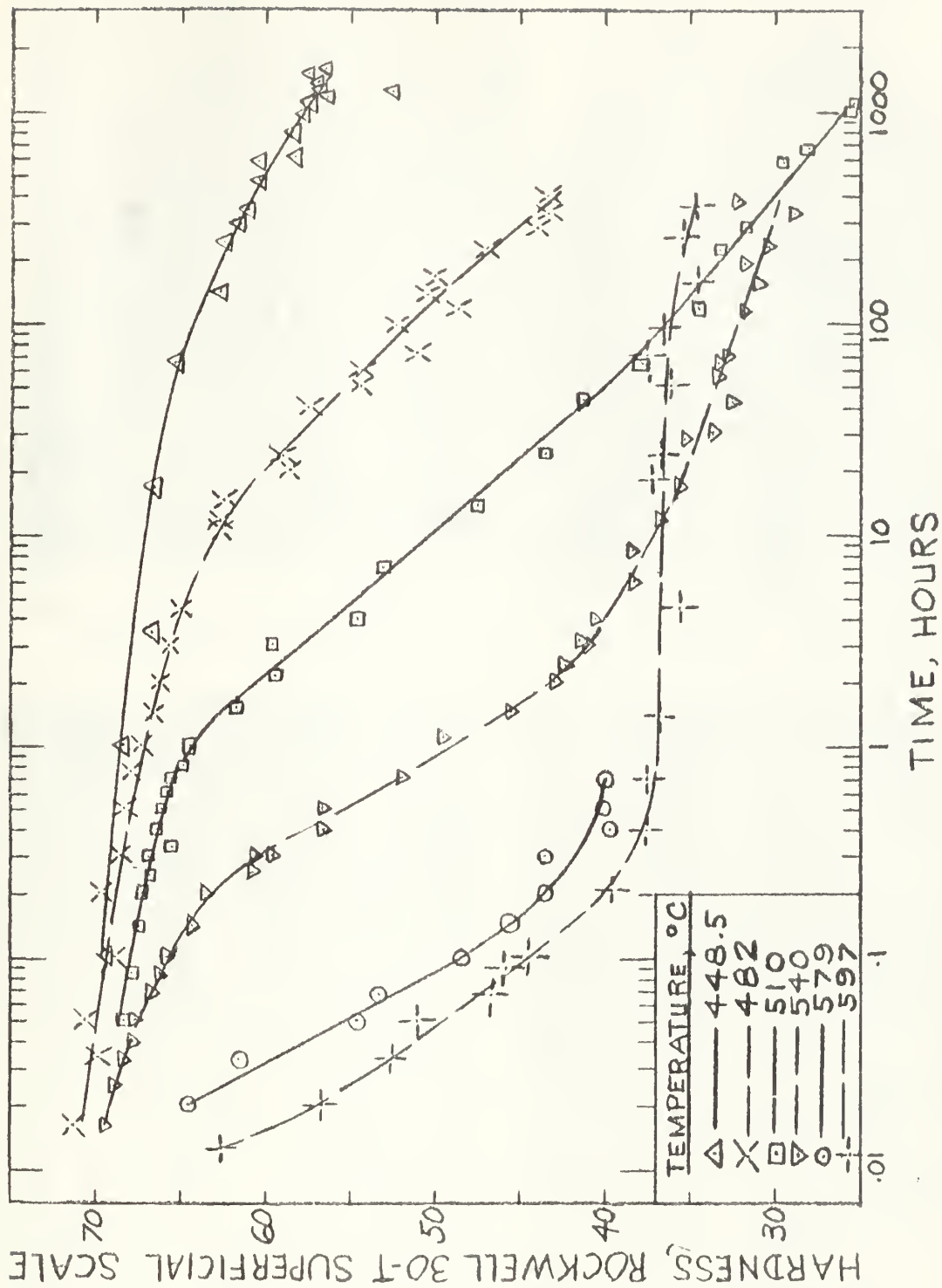


FIG.4 THE EFFECT OF ANNEALING TIME AT VARIOUS TEMPERATURES ON THE ROOM TEMPERATURE HARDNESS FOR A 60 PER CENT COLD-ROLLED VACUUM MELTED HIGH PURITY IRON



FIG.5 AS ROLLED CONDITION
(ROCKWELL 30T 70)

FIG.6 AT 540°C FOR 1 MINUTE
(ROCKWELL 30T 69.2)



FIG.7 AT 540°C FOR 6 MINUTES
(ROCKWELL 30T 65.9)

FIG.8 AT 540°C FOR 18 MINUTES
(ROCKWELL 30T 59.4)

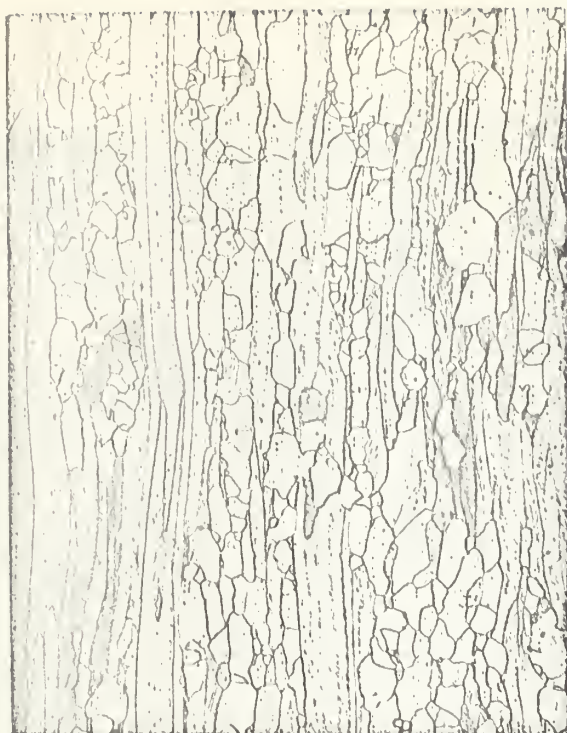


FIG. 9 AT 540°C FOR 42 MINUTES (ROCKWELL 30T 51.9) -150X- FIG. 10 AT 540°C FOR 3 HOURS (ROCKWELL 30T 41)



FIG. 11 AT 540°C FOR 30 HOURS (ROCKWELL 30T 33.7) -150X- FIG. 12 AT 540°C FOR 329.6 HOURS (ROCKWELL 30T 28.8)



— Roll Direction —



FIG.13 AT 597°C FOR 2 MINUTES (ROCKWELL 30T 52.6) -150 X- FIG.14 AT 540°C FOR 42 MINUTES (ROCKWELL 30T 51.9)



FIG.15 AT 482°C FOR 96 HOURS (ROCKWELL 30T 52.4) -150 X- FIG.16 AT 448.5°C FOR 1242 HOURS (ROCKWELL 30T 52.5)

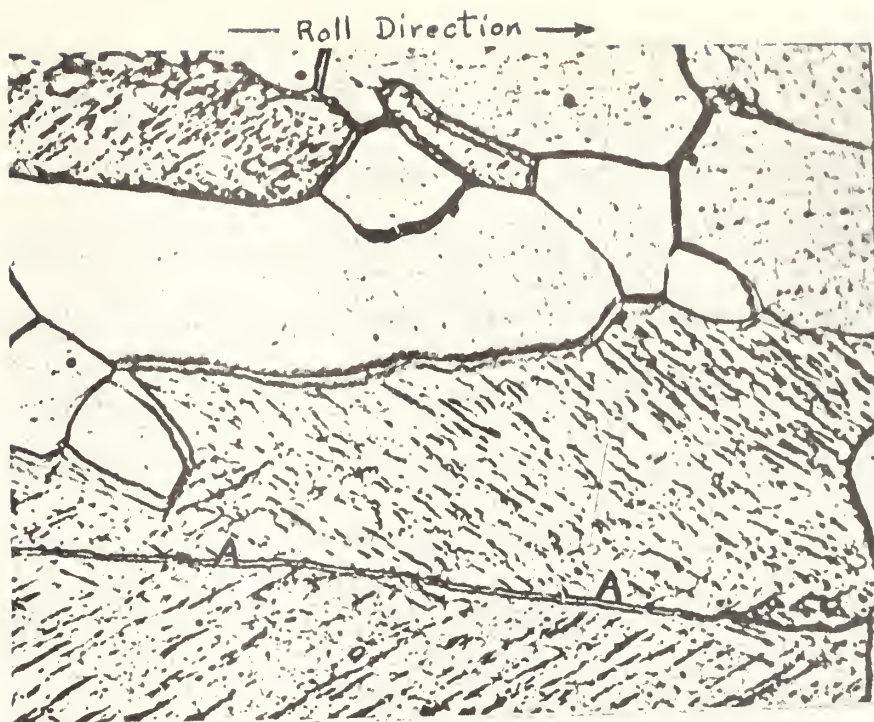


FIG.17 AT 597°C FOR 2 MINUTES
(ROCKWELL 30T 52.6)
-2000 X-



FIG.18 AT 448.5°C FOR 1242 HOURS
(ROCKWELL 30T 52.5)
-2000X-

B. Dynamic Study Results

Fig. 19 essentially achieves the basic goal of this investigation in that it shows the effect of simultaneous strain on the softening behavior of the test material at the annealing temperatures of 540°C and 482°C . The solid-lined static curves, obtained from no-load conditions, are the same as those of Fig. 4 for the corresponding temperatures. The dashed curves show the effect of imposing the simultaneous constant strain rates of .005% per hour and .6% per hours.

It is noted in Fig. 19 that at the annealing temperature of 540°C the effect of the simultaneous imposition of the slower strain rate of .005% per hour was to slightly accelerate the softening of the test material throughout the anneal. The deviation, however, falls approximately within the scatter shown in Fig. 4. The effect of imposing the faster strain rate of .6% per hour was to first accelerate the softening and then retard the softening with an end result of a harder annealed material. Both of the preceding observations were made in comparison with the static curve at 540°C . Figs. 20 through 28 are photomicrographs at 500 magnification of the microstructures of the test material at various points along the static and dynamic 540°C annealing curves of Fig. 19. It is noted in Figs. 20 through 22, for the static anneal at 540°C , that recrystallization is in progress and increases with time-at-temperature. Little, if any, polygonization of the deformed grains is in evidence throughout the annealing period. The test material apparently had softened by typical recovery and recrystallization.

Figs. 23 through 25 show the progress of the faster dynamic anneal, again at 540°C . In Fig. 23 polygonization is evident in the deformed grains after only 18 minutes of anneal time. A comparison of Figs 20 and 23 show that the degree of recrystallization for the static anneal is significantly greater than that of the dynamic anneal for the same time. The lower softness of the latter specimen must then arise as a result of the structural difference within the original deformed grains, for the greater number of new strain-free grains alone, as shown in Fig. 20, should result in a lower softness for the static specimen. An examination of the residual deformed grains supports this contention. It may be seen in Fig. 23 that the boundaries between the deformed grains are wavy and jagged. The regions within some of the deformed grains, on close examination, may be seen to consist of a substructure. Both of these appearances are associated with polygonization. These characteristics are absent in Fig. 20 where the deformed grains still show the more or less regular boundaries containing the diffused bands indicative of normal recovery. Thus, the removal of the strained regions by polygonization more than offsets the smaller amount of recrystallization as compared to the static specimen in so far where room temperature hardness measurements are concerned. Figs. 24 and 25 show that polygonization has progressed still further in the original deformed grains of the specimens subject to the fast strain rate, resulting in a composite polygonized and recrystallized microstructure after 30 hours at 540°C .

It is interesting to note in Fig. 19 that the faster rate dynamic

curve crosses that of the static at about 1.5 hours of anneal time at 540°C and that the hardness after 30 hours is about six Rockwell 30T points greater than that of the static. The aforementioned curve cross-over may be explained with reference to Figs. 20 to 22 as compared to Figs. 23 to 25. The increased recrystallization with time under static conditions more than offsets the softening as a result of the combination of recrystallization and polygonization of the dynamic specimens. Finally, at 30 hours the static specimen is virtually all recrystallized. In contrast, the dynamic specimen consists of a combination of recrystallized and well defined polygonized grains. Furthermore, some polygonization has occurred in the recrystallized grains due to the straining. It is well known that for a strain-free condition hardness increases with grain fineness. The combined recrystallized and polygonized dynamic specimen of Fig. 25 may be said to contain an average smaller grain size than for the corresponding static specimen of Fig. 22.

Figs. 26 through 28 show the microstructures of the static, slow dynamic, and fast dynamic specimens after a 30-hour anneal at 540°C . The varying degrees of polygonization are self evident. An examination of the specimens for annealing times of less than 30 hours showed no apparent difference between the microstructures of the static and slow-strain-rate dynamic specimens. At 30 hours for the latter test condition it may be seen in Fig. 27 that a very slight amount of polygonization is evident. Otherwise, here again the microstructure is very similar to that of the static and one would expect essentially the same hardness,

as was obtained.

The processes of both high temperature deformation and polygonization occur by dislocation glide and climb. High temperature deformation of an initially recrystallized state frequently results in polygonization. (8) The strain is initially accounted for by the formation and glide of dislocations which subsequently climb by thermal activation and form polygonized boundaries. Since glide, as well as climb, is necessary for polygonization one must conclude that the slow strain-rate and associated small amount of glide was ineffective in accelerating polygonization sufficiently to offset the rapid recrystallization occurring at 540°C . A strain rate of approximately 100-fold greater, however, was sufficient to attain the necessary glide and climb.

In Fig. 19 it can be seen that the effect of the imposition of simultaneous strain rate at the 482°C anneal temperature is quite different from that at the 540°C temperature for the fast strain rates. The softening behavior of the test material has been accelerated by both the slow and fast strain rates at the 482°C temperature. However, in contrast to the results for the higher temperature the spread in hardness between the static and fast strain rate curves continuously increases with time. This accelerated softening is again due to the presence of polygonization in the dynamic specimens of the test material and may be best explained with reference to the corresponding microstructures.

Figs. 29 through 31 show the slow progress of recrystallization in the static specimens at the 482°C anneal. Figs. 32 through 34, which

represent the fast strain rate, when compared with Figs. 29 through 31, clearly evidence the progress of polygonization and the delayed recrystallization of specimens subjected to the fast strain rate while at the same temperature. It is quite apparent from observations of Figs. 35 through 37, which show, respectively, the microstructures of the static, and the slow and fast dynamic specimens after 49 hours at 482°C , that the differences in hardness may be explained by the relative predominance of recrystallization and unsoftened deformed grains on one hand and recrystallization and polygonized deformed grains on the other hand. Fig. 35 shows approximately 50% of the area consists of recrystallized grains with the balance in unsoftened deformed grains. Fig. 36 shows less than 10% recrystallization with the balance largely in the early and intermediate stages of polygonization. Fig. 37 shows a well defined predominance of polygonization with some localized recrystallization.

The normal static anneal curve of 482°C in Fig. 19, as compared with that at 540°C , shows that recrystallization is delayed due to the lower temperature. This in itself is a commonly accepted result, as shown in the literature on recrystallization kinetics. (2) It is this delay of recrystallization due to the lower anneal temperature that has enabled polygonization, when accelerated or induced by dynamic strain, to more effectively replace the deformed grain structure over a given time period, resulting in an overall greater softening after 49 hours. However, it would be expected that a curve crossover, as was obtained at 540°C ,

would result after longer times due to the amount of recrystallization in the static specimen. Furthermore, it is of interest to note that the recrystallization process is sufficiently slow at this lower temperature such that even with the slow strain rate polygonization can progress sufficiently so as to be competitive with recrystallization, as may be seen in Figs. 35 and 36. In the latter case polygonization has gained precedence over recrystallization in some of the original deformed grains.

Thus, the results of the dynamic study show that the imposition of simultaneous plastic strain affected the softening behavior, not by altering the recrystallization process per se, but by accelerating a competing process. This competing process is polygonization. The precedence of the competing mechanism is highest under conditions where recrystallization is slowest, such as at low temperatures and, undoubtedly, also for small amounts of cold work. At higher temperatures higher strain rates are necessary to further accelerate the competing polygonization process in order for it to take precedence over recrystallization. Whether or not the dynamic strain will result in a harder or softer structure when polygonization takes place will depend upon the degree of recrystallization that has taken place in the static specimen annealed for the same length of time. In the extreme case, a completely recrystallized structure would have a much coarser grain size than that of a completely polygonized structure, and accordingly the former should be softer than the latter structure.

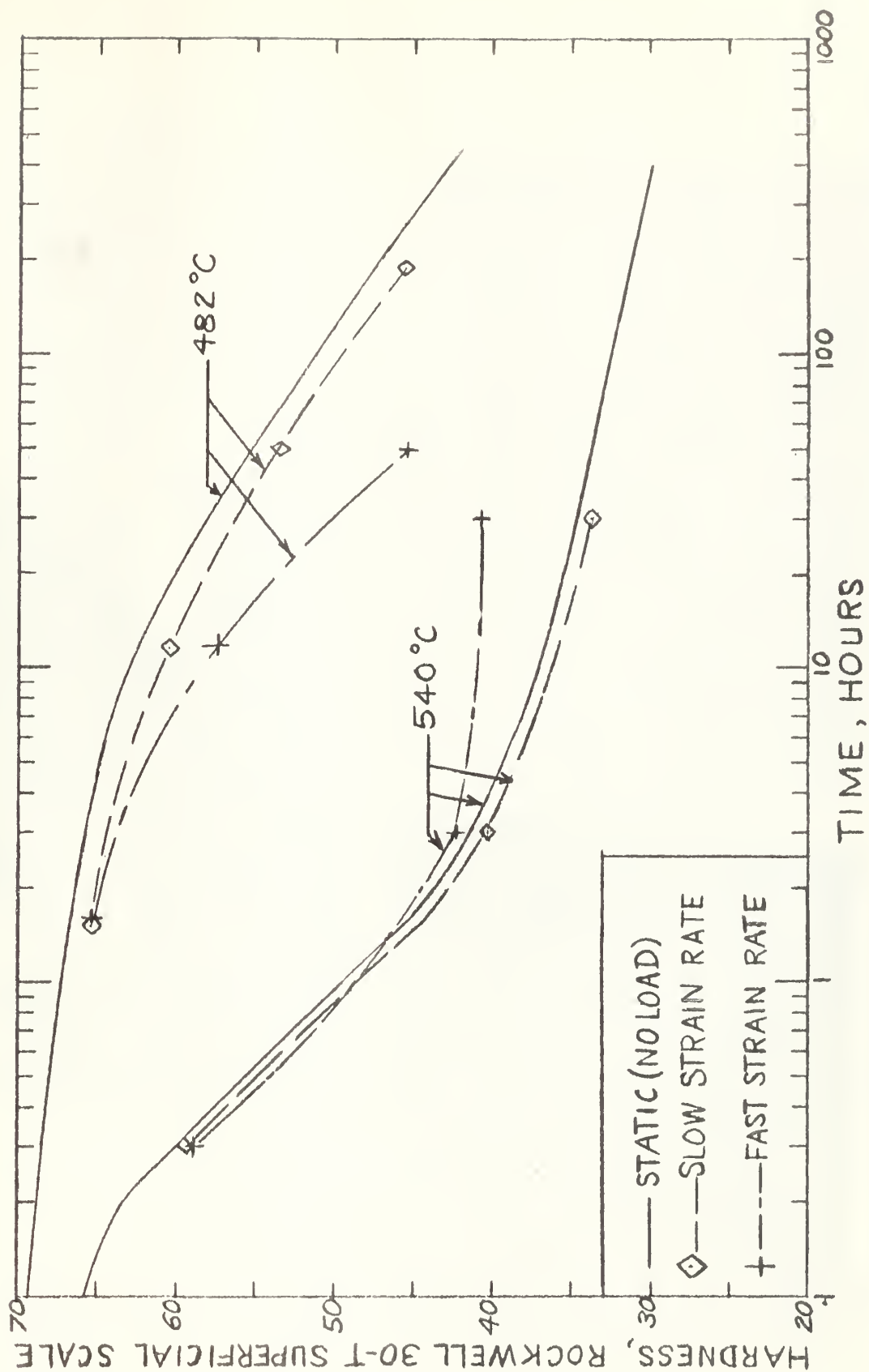


FIG.19 THE EFFECT OF SIMULTANEOUS STRAIN ON THE SOFTENING BEHAVIOR OF A 60 PER CENT COLD-ROLLED VACUUM-MELTED HIGH PURITY IRON



FIG.20 AT 540°C
FOR 18 MINUTES
(ROCKWELL 30T 59.4)

-500X-

— Roll Direction →



FIG.21 AT 540°C FOR 3 HOURS
(ROCKWELL 30T 41)



FIG.22 AT 540°C FOR 30 HOURS
(ROCKWELL 30T 33.7)



FIG.23 AT 540°C, UNDER
 .6%/HR. STRAIN RATE, FOR
 18 MINUTES
 (ROCKWELL 30T 58.9)

-500X-

Roll Direction →

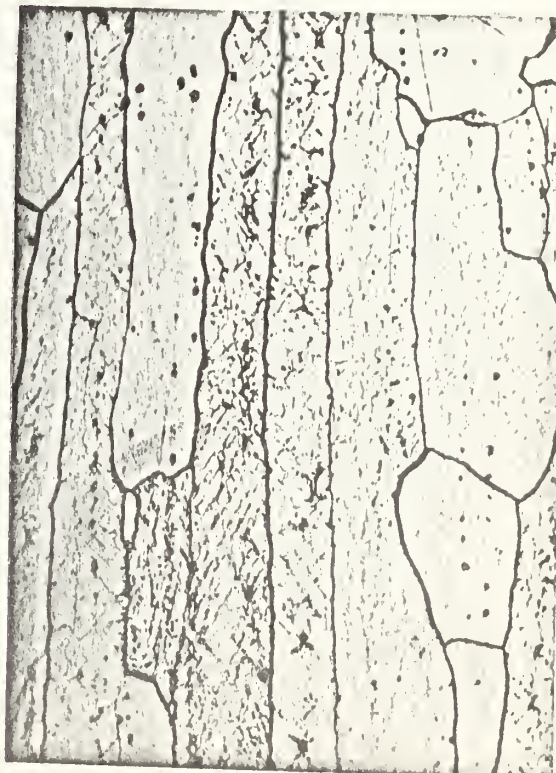


FIG.24 AT 540°C, UNDER .6%/HR. STRAIN
 RATE, FOR 3 HOURS (ROCKWELL 30T 42.1)



FIG. 25 AT 540°C, UNDER .6%/HR. STRAIN
 RATE, FOR 30 HOURS (ROCKWELL 30T 40.7)

FIG. 26 AT 540°C FOR
30 HOURS
(ROCKWELL 30T 33.7)



-500 X- -Roll Direction→



FIG. 27 AT 540°C, UNDER .005%/HR.
STRAIN RATE, FOR 30 HOURS
(ROCKWELL 30T 33.6)



FIG. 28 AT 540°C, UNDER .6%/HR.
STRAIN RATE, FOR 30 HOURS
(ROCKWELL 30T 40.7)



FIG. 29 AT 482°C FOR
1.5 HOURS
(ROCKWELL 30T 66.9)

-500 X- — Roll Direction —→



FIG. 30 AT 482°C FOR 11.7 HOURS
(ROCKWELL 30T 62.7)



FIG. 31 AT 482°C FOR 49 HOURS
(ROCKWELL 30T 54.3)



FIG. 32 AT 482°C, UNDER
6%/HR. STRAIN RATE,
FOR 1.5 HOURS
(ROCKWELL 30T 65.3)

-500 X-

— Roll Direction —→



FIG. 33 AT 482°C, UNDER .6%/HR. STRAIN
RATE, FOR 11.7 HOURS (ROCKWELL 30T 57.4)



FIG. 34 AT 482°C, UNDER .6%/HR. STRAIN
RATE, FOR 49 HOURS (ROCKWELL 30T 45.4)

FIG. 35 AT 482°C FOR
49 HOURS
(ROCKWELL 30T 54.3)



— 500 X — — Roll Direction —→



FIG. 36 AT 482°C, UNDER .005%/HR.
STRAIN RATE, FOR 49 HOURS
(ROCKWELL 30T 53.5)



FIG. 37 AT 482°C, UNDER .6%/HR. STRAIN
RATE, FOR 49 HOURS (ROCKWELL 30T 45.4)

5. Summary of Results and Conclusions

A. Static and dynamic annealing curves for a number of temperatures were obtained for a 60% cold-worked vacuum-melted high purity iron.

B. Parallelism between the relative drop in hardness and percent recrystallization was obtained for the static annealing at 540°C . Static annealing curves of a high purity metal may be somewhat modified from those of a lower purity metal due to the presence of a competing process. This process was shown to be polygonization which plays a larger role with decreasing temperature.

C. The effect of simultaneous plastic strain during annealing modifies the softening behavior by accelerating the polygonization process. The degree of acceleration is dependent upon the strain rate, temperature, and time. The degree of softening obtained, as compared to a static anneal for the same temperature and time, depends on the ease and amount of recrystallization of the static annealed material.

ACKNOWLEDGEMENTS

It is a pleasure to acknowledge the generous assistance and encouragement of Associate Professor A. Goldberg. The author is also indebted to the following members of the metallurgy laboratory staff; D. Clark for his valuable assistance in specimen preparation, R. F. Edwards for equipment aid, and R. E. Meyers and R. Smith (electronics laboratory staff member) for their assistance in the design of the constant strain rate units. Last but not least, the author would like to acknowledge the encouragement and patience of his wife, Barbara, throughout the course of this investigation.

BIBLIOGRAPHY

1. M. B. Bever, On the Thermodynamics and Kinetics of Recovery, Creep and Recovery, ASM, pp. 14-51, 1957.
2. J. E. Burke and D. Turnbull, Recrystallization and Grain Growth, Progress in Metal Physics 3, Interscience Publishers, pp. 220-292, 1952.
3. E. C. W. Perryman, Recovery of Mechanical Properties, Creep and Recovery, ASM, pp. 111-145, 1957.
4. O. D. Sherby, A. Goldberg and J. E. Dorn, Effect of Prestrain Histories on the Creep and Tensile Properties of Aluminum, Trans. ASM, 46, pp. 681-700, 1954.
5. N. J. Grant and A. G. Bucklin, Creep-Rupture and Recrystallization of Monel from 700 to 1700°F, Trans. ASM, 45, pp. 151-170, 1953.
6. R. F. Mehl, Recrystallization, Metals Handbook, ASM, pp. 259-263, 1948.
7. A. Goldberg, Private Communication
8. C. Crussard and R. Tamhankar, High-Temperature Deformation of Steels A Study of Equicohesion, Activation Energies, and Structural Modifications, Trans. Metallurgical Society of AIME, 212, pp. 718-730, 1958.
9. M. J. Talbot, Etude De La Polygonisation Du Fer De "Zone Fondue" Competition Entre La Polygonisation Et La Recristallisation. Cas D'Autres Metaux, Unpublished Work.
10. R. W. Cahn, Internal Strains and Recrystallization, Progress in Metal Physics 2, Interscience Publishers, pp. 151-175, 1950.
11. I. I. Betcherman, Rate Processes in Physical Metallurgy, Progress in Metal Physics 2, Interscience Publishers, pp. 53-89, 1950.
12. A. Guinier and F. Tennevin, Researches on the Polygonization of Metals, Progress in Metal Physics 2, Interscience Publishers, pp. 177-192, 1950.
13. C. Crussard, F. Aubertin, B. Faoul and G. Wyon, Polygonization in Strongly Deformed Metals, Progress in Metal Physics 2, Interscience Publishers, pp. 193-202, 1950.





thesG867

Investigation of the effect of simultane



3 2768 002 13603 8
DUDLEY KNOX LIBRARY